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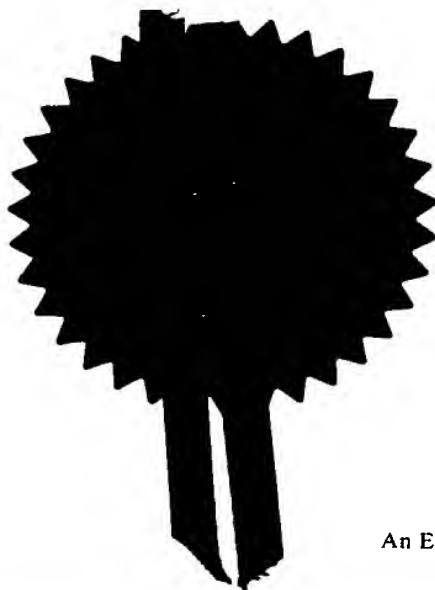
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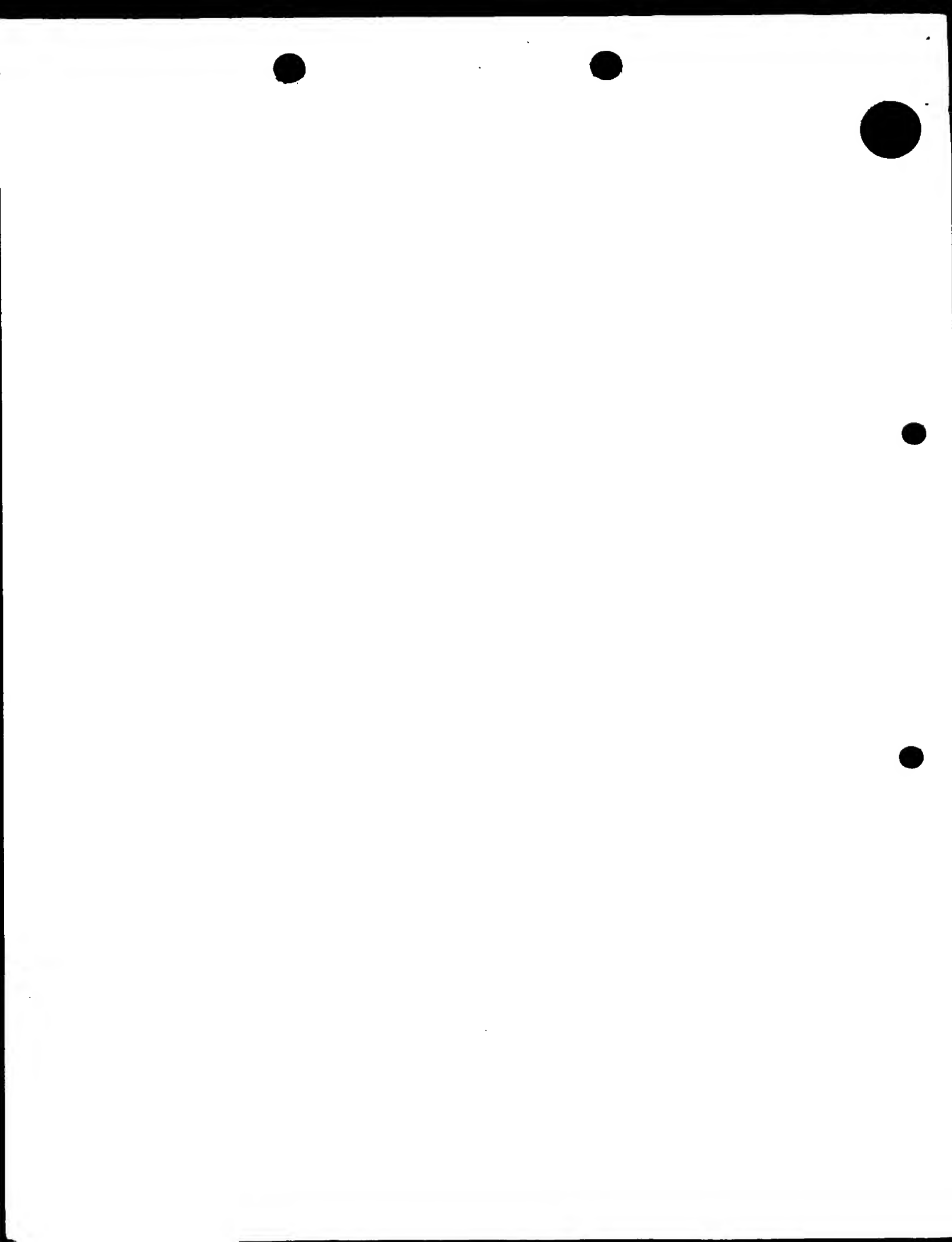
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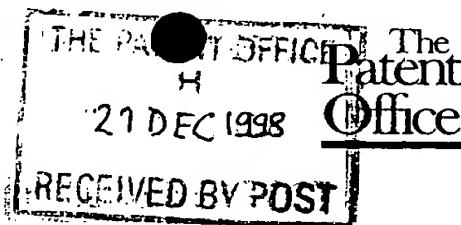
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Signed *Andrew Gersay*
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21 DEC 98 E412990-1 002944
F01/7700 0.00 - 9827894.8

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1. Your reference	SMC 60331		
2. Patent application number (The Patent Office will fill in this part)	9827894.8		
3. Full name, address and postcode of the or of each applicant (underline all surnames)	Zeneca Limited 15 Stanhope Gate London W1Y 6LN		
Patents ADP number (if you know it)	6254007002		
If the applicant is a corporate body, give the country/state of its incorporation	United Kingdom		
4. Title of the invention	Composition		
5. Name of your agent (if you have one)	MAYALL, John		
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Patents ADP number (if you know it)	6244313002		
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7. If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application	Number of earlier application		Date of filing (day / month / year)
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Description

23

Claim(s)

2

Abstract

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Statement of inventorship and right to grant of a patent (*Patents Form 7/77*)

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Date 18/12/98

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COMPOSITION

This invention relates to coloured water-dissipatable polymers, in particular acrylic water-dissipatable polymers and to inks containing coloured water-dissipatable polymers and to their use in ink jet printing.

5 Ink jet printing methods involve a non-impact printing technique for printing an image onto a substrate using ink droplets ejected through a fine nozzle onto a substrate without bringing the nozzle into contact with the substrate.

10 There are many demanding performance requirements for colorants and inks used in ink jet printing. For example they desirably provide sharp, non-feathered images having good water-fastness, light-fastness and optical density. The inks are often required to dry quickly when applied to a substrate to prevent smudging, but they should not form a crust over the tip of an ink jet nozzle because this will stop the printer from working. The inks should also be stable to storage over time without decomposing or forming a precipitate which could block the fine nozzle. The most popular ink jet printers are the thermal and piezoelectric ink jet printers.

15 There is a need for inks which are suitable for both thermal and piezo ink jet printers, have high colour strength and produce images having a high light-fastness and water-fastness when printed on a substrate.

20 We have surprisingly found that the coloration of water-dissipatable polymers carrying pendant hydroxy functional groups by reacting the hydroxy functional groups with a colorant, a bridging group for a colorant or a colorant precursor and further reaction of the bridging compound or colorant precursor to give a coloured polymer results in a coloured water-dissipatable polymer which is suitable for use in inks for thermal and piezo ink jet printers.

25 According to a first aspect of the present invention there is provided a water-dissipatable polymer having colorant attached thereto through a covalent -O- link.

The polymer may be any water-dissipatable polymer with hydroxy functional groups. Examples include but are not limited to acrylic polymers, acrylic copolymers, polyesters and polyurethanes that include functional hydroxy groups. Preferably acrylic polymers (which include acrylic copolymers) are used.

30 According to a further aspect of the present invention there is provided a water-dissipatable acrylic polymer having colorant attached thereto through a covalent -O- link.

A -O- link may be exemplified by but is not limited by the following links such as ether = R-O-R, ester = R-O-C(O)-R and phosphate ester = R-O-P(O)(OH)-.

35 Preferably the number average molecular weight (M_n) of the water-dissipatable polymer is less than 25,000, more preferably is less than 20,000, especially less than

15,000. The Mn of the polymer may be measured by gel permeation chromatography ("gpc").

5 The gpc method used for determining Mn preferably comprises applying the acrylic polymer to a chromatography column packed with cross-linked polystyrene/divinyl benzene, eluting the column with tetrahydrofuran at a temperature of 40°C and assessing the Mn of the acrylic polymer compared to a number of a polystyrene standards of a known Mn. Suitable cross-linked polystyrene/divinyl benzene chromatography columns are commercially available from Polymer Laboratories.

10 If the gpc method for determining Mn does not work for any reason, for example the polymer has an unexpected interaction with the gpc column give an unrealistic result, the Mn may be determined using alternative methods, for example by multi-angle light scattering (MALLS).

15 The water-dissipatable polymer has preferably been obtained from the polymerisation of one or more olefinically unsaturated monomers having water dispersing groups, and one or more olefinically unsaturated monomers having hydroxyl functional groups optionally in the presence of one or more olefinically unsaturated monomers which are free from water dispersing groups.

20 Preferred olefinically unsaturated monomers having hydroxy functional group(s) include, but are not limited to, hydroxy ethyl methacrylate, hydroxy ethyl acrylate, hydroxy propyl methacrylate, hydroxy propyl acrylate, hydroxybutyl methacrylate, hydroxy polyethylene glycol methacrylates, N-methylol acrylamide, hydroxy polypropylene methacrylates, glycidyl methacrylate, TONE M100 (available from Union Carbide).

25 Olefinically unsaturated monomers having a group which is convertible to a reactive hydroxy functional group include but are not limited to vinyl acetate, vinyl benzoate, vinyl benzyl chloride, vinyl bromide and vinyl chloride.

30 Dispersing groups provide the facility of self-dispersibility and solubility to the polymer in ink media, especially in water. The dispersing groups may be ionic, non-ionic or a mixture of ionic and non-ionic dispersing groups. Preferred ionic dispersing groups include basic amine groups, cationic quaternary ammonium groups and acid groups, for example phosphoric acid groups, sulphonic acid groups and carboxylic acid groups.

35 The dispersing groups may be incorporated into the polymer in the form of monomers or oligomers bearing the appropriate dispersing groups. One may also react a polymer which is not water-dissipatable with monomers or oligomers which make the polymer water-dissipatable.

The acid groups may be subsequently fully or partially neutralised with a base containing a cationic charge to give a salt. If the acid dispersing groups are used in combination with a non-ionic dispersing group, neutralisation may not be required. The conversion of any free acid groups into the corresponding salt may be effected during

the preparation of the water-dissipatable polymer and/or during the preparation of an ink from the water-dissipatable polymer.

Preferably the base used to neutralise any acid dispersing groups is ammonia, an amine or an inorganic base. Suitable amines are tertiary amines, for example triethylamine or triethanolamine. Suitable inorganic bases include alkaline hydroxides and carbonates, for example lithium hydroxide, sodium hydroxide, or potassium hydroxide. A quaternary ammonium hydroxide, for example $N^+(CH_3)_4OH^-$, can also be used. Generally a base is used which gives the required counter ion desired for the ink which is prepared from the polymer. For example, suitable counter ions include Li^+ , Na^+ , K^+ , NH_4^+ and substituted ammonium salts.

Preferred olefinically unsaturated monomers providing ionic dispersing groups include acrylic acid, methacrylic acid, itaconic acid, maleic acid, monoalkyl itaconates (for example, monomethyl maleate, monoethyl maleate, monobutyl maleate and monooctyl maleate), citraconic acid, styrenesulfonic acid, vinylbenzylsulfonic acid, vinylsulfonic acid, acryloyloxyalkyl sulfonic acids (for example, acryloyloxymethyl sulfonic acid, acryloyloxyethyl sulfonic acid, acryloyloxypropyl sulfonic acid and acryloyloxybutyl sulfonic acid), methacryloyloxymethyl sulfonic acid, methacryloyloxyethyl sulfonic acid, methacryloyloxypropyl sulfonic acid and methacryloyloxybutyl sulfonic acid), 2-acrylamido-2-alkylalkane sulfonic acids (for example, 2-acrylamido-2-methylethanesulfonic acid, 2-acrylamido-2-methylpropanesulfonic acid and 2-acrylamido-2-methylbutane sulfonic acid), 2-methacrylamido-2-alkylalkane sulfonic acids (for example, 2-methacrylamido-2-methylethanesulfonic acid, 2-methacrylamido-2-methylpropanesulfonic acid and 2-methacrylamido-2-methylbutanesulfonic acid), mono-(acryloyloxyalkyl)phosphates (for example, mono(acryloyloxyethyl)phosphate and mono(3-acryloyloxypropyl)phosphates) and mono(methacryloyloxyalkyl)phosphates (for example, mono(methacryloyloxyethyl)phosphate and mono(3-methacryloyloxypropyl)phosphate).

Non-ionic dispersing groups may be in-chain, pendant or terminal groups. Preferably non-ionic dispersing groups are pendant polyoxyalkylene groups, more preferably polyoxyethylene groups. The non-ionic groups may be introduced into the water-dissipatable polymer in the form of a compound bearing non-ionic dispersing groups and at least one (although preferably only one) copolymerisable olefinically unsaturated group.

Preferred olefinically unsaturated monomers providing non-ionic dispersing groups include alkoxy polyethylene glycol (meth)acrylates, preferably having a number average molecular weight of from 350 to 2000. Examples of such monomers which are commercially available include ω -methoxypolyethylene glycol acrylate (mean polymerisation degree of polyethylene glycol is about 9) and diethylene glycol vinyl ether.

The nature and level of dispersing groups in the polymer influences whether a solution, dispersion, emulsion or suspension is formed on dissipation of the water-dissipatable polymer.

5 The dispersing group content of the water-dissipatable polymer may vary within wide limits but is preferably sufficient to make the water-dissipatable polymer form stable ink-jet printing inks in water and aqueous media.

10 In addition to the water-dissipatable polymer comprising olefinically unsaturated monomers having a reactive hydroxy functional group or a group which is convertible to a reactive hydroxy functional group, and one or more olefinically unsaturated monomers having water dispersing groups, the water-dissipatable polymer may also contain one or more olefinically unsaturated monomers which are free from water dispersing groups and/or reactive groups.

15 Preferred olefinically unsaturated monomers which are free from dispersing groups include alkyl(meth)acrylates, optionally substituted styrenes, methacrylamides, allyl compounds, dienes, vinyl ethers, vinyl ketones, vinyl halides, vinylidene halides, olefins and unsaturated nitriles.

20 Preferred alkyl(meth)acrylates contain less than twenty carbon atoms. Examples include methyl acrylate, ethyl acrylate, n-propyl acrylate, isopropyl acrylate, n-butyl acrylate, isobutyl, sec-butyl acrylate, amyl acrylate, hexyl acrylate, 2-ethylhexyl acrylate, octyl acrylate, tert-octyl acrylate, 2-phenoxyethyl acrylate, 2-chloroethyl acrylate, 2-bromoethyl acrylate, 4-chlorobutyl acrylate, cyanoethyl acrylate, 2-acetoxyethyl acrylate, dimethylaminoethyl acrylate, benzyl acrylate, methoxybenzyl acrylate, 2-chlorocyclohexyl acrylate, acrylate, cyclohexyl acrylate, furfuryl acrylate, tetrahydrofurfuryl acrylate, phenyl acrylate, 5-hydroxypentyl acrylate, 2,2-dimethyl-3-hydroxypropyl acrylate, 2-methoxyethyl acrylate, 3-methoxybutyl acrylate, 2-ethoxyethyl acrylate, 2-isopropoxyethyl acrylate, 2-butoxyethyl acrylate, 2-(2-methoxyethoxy)ethyl acrylate, 2-(2-butoxyethoxy)ethyl acrylate, 1-bromo-2-methoxyethyl acrylate, 1,1-dichloro-2-ethoxyethyl acrylate, methyl methacrylate, ethyl methacrylate, n-propyl methacrylate, isopropyl methacrylate, n-butyl methacrylate, isobutyl methacrylate, sec-butyl methacrylate, amyl methacrylate, hexyl methacrylate, cyclohexyl methacrylate, benzylmethacrylate, chlorobenzyl methacrylate, octyl methacrylate, N-ethyl-N-phenylaminoethyl methacrylate, 2-(3-phenylpropyloxy)ethyl methacrylate, dimethylaminophenoxyethyl methacrylate and furfuryl methacrylate. Aromatic examples include but are not limited to 4-alkyl phenylacrylate or methacrylate, phenyl methacrylate, phenyl acrylate, and β -naphthyl methacrylate, 35

40 Preferred optionally substituted styrenes include styrene, methylstyrene, dimethylstyrene, trimethylstyrene, ethylstyrene, diethylstyrene, isopropylstyrene, butylstyrene, hexylstyrene, cyclohexylstyrene, decylstyrene, chloromethylstyrene, trifluoromethylstyrene, ethoxymethylstyrene, acetoxymethylstyrene, methoxystyrene, 4-methoxy-3-methylstyrene, dimethoxystyrene, chlorostyrene, dichlorostyrene,

trichlorostyrene, tetrachlorostyrene, pentachlorostyrene, bromostyrene, dibromostyrene, iodostyrene, trifluorostyrene and 2-bromo-4-tri-fluoromethylstyrene.

Preferred methacrylamides contain less than 12 carbon atoms. Examples include methylmethacrylamide, tert-butylmethacrylamide, tert-octylmethacrylamide, benzylmethacrylamide, cyclohexylmethacrylamide, phenylmethacrylamide, dimethylmethacrylamide, dipropylmethacrylamide, hydroxyethyl-N-methylmethacrylamide, N-methylphenylmethacrylamide, N-ethyl-N-phenylmethacrylamide and methacrylhydrazine.

Preferred allyl compounds include allyl acetate, allyl caproate, allyl caprylate, allyl laurate, allyl palmitate, allyl stearate, allyl benzoate, allyl acetoacetate, allyl lactate, allyloxyethanol, allyl butyl ether and allyl phenyl ether.

Preferred vinyl ethers contain less than 20 carbon atoms. Examples include methyl vinyl ether, butyl vinyl ether, hexyl vinyl ether, octyl vinyl ether, decyl vinyl ether, ethylhexyl vinyl ether, methoxyethyl vinyl ether, ethoxyethyl vinyl ether, chloroethyl vinyl ether, 1-methyl-2,2-dimethylpropyl vinyl ether, 2-ethylbutyl vinyl ether, hydroxyethyl vinyl ether and dimethylaminoethyl vinyl ether.

Preferred vinyl ketones contain less than 12 carbon atoms. Examples include methyl vinyl ketone, phenyl vinyl ketone and methoxyethyl vinyl ketone.

Preferred vinyl halides include vinyl chloride, vinylidene chloride and chlorotrifluoro ethylene.

Preferred olefins include unsaturated hydrocarbons having less than 20 carbon atoms. Examples include dicyclopentadiene, ethylene, propylene, 1-butene, 1-pentene, 1-hexene, 4-methyl-1-pentene, 1-heptene, 1-octene, 1-decene, 5-methyl-1-nonene, 5,5-dimethyl-1-octene, 4-methyl-1-hexene, 4,4-dimethyl-1-pentene, 5-methyl-1-hexene, 4-methyl-1-heptene, 5-methyl-1-heptene, 4,4-dimethyl-1-hexene, 5,5,6-trimethyl-1-heptene, 1-dodecene and 1-octadecene.

Preferred unsaturated nitriles include acrylonitrile and methacrylonitrile

The preferred olefinically unsaturated monomers which are free from dispersing groups are the alkyl (meth)acrylates containing less than 20 carbon atoms, especially those specifically listed above.

When the water-dissipatable polymer is prepared by the polymerisation of (a) olefinically unsaturated monomers providing dispersing groups in the presence of (b) olefinically unsaturated monomers which are free from dispersing groups and in the presence of (c) olefinically unsaturated monomers having hydroxy functional groups or a group which is convertible to a reactive hydroxy functional group, it is preferred that the amount of (b) is from 1 to 95%, more preferably from 2 to 90% and the amount of (c) is from 1 to 95%, more preferably from 5 to 90% by weight relative to the weight of (a) + (b) + (c).

The water-dissipatable polymer preferably has an acid value of from 0 to 750mgKOH/g, more preferably 50 to 450mgKOH/g, especially 50 to 225mgKOH/g.

The water-dissipatable polymer may be prepared in a conventional manner by polymerising the olefinically unsaturated monomers providing dispersing groups and olefinically unsaturated monomers having a reactive hydroxy functional group or a group which is convertible to a reactive hydroxy functional group, either alone or in the presence of olefinically unsaturated monomers which are free from dispersing and reactive hydroxy functional groups. Temperatures of from 20°C and 180°C are preferred. The polymerisation may be continued until reaction between the monomers is complete.

In one embodiment the water-dissipatable polymer may be prepared by polymerising an acrylic oligomer having water dispersing groups and one olefinically unsaturated terminal group in the presence of one or more olefinically unsaturated monomers having a reactive hydroxy functional group or a group which is convertible to a reactive hydroxy functional group, and one or more olefinically unsaturated monomers which are free from water dispersing groups and/or olefinically unsaturated monomers having water dispersing groups.

Alternatively an acrylic oligomer which is free from water dispersing groups may be polymerised in the presence of one or more olefinically unsaturated monomers having a reactive hydroxy functional group or a group which is convertible to a reactive hydroxy functional group and one or more olefinically unsaturated monomers having water dispersing groups.

Preferred polymerisation methods include solution polymerisation, emulsion polymerisation, suspension polymerisation and solution/dispersion polymerisation and such general methods as are well known in the art. More preferably aqueous or non-aqueous solution polymerisation and emulsion polymerisation is used and most preferably solution polymerisation is used.

If desired, an initiator may be used to assist acrylic polymer formation. Suitable initiators are free-radical generators. Examples of catalysts include azobis compounds, peroxides, hydroperoxides, redox catalysts, etc., for example, potassium persulfate, ammonium persulfate, tert-butyl peroctoate, benzoyl peroxide, isopropyl percarbonate, 2,4-dichlorobenzoyl peroxide, methyl ethyl ketone peroxide, cumene hydroperoxide, dicumyl peroxide, azobisisobutyronitrile, azobis(2-amidino-propane)hydrochloride and the like.

Typically 0.05 to 5% by weight of initiator is used relative to the total weight of the monomers. Preferably the polymerisation is performed in the presence of an emulsifying agent.

The Mw of the acrylic polymer may be controlled by the addition of chain transfer agents and/or through the adjustment of the ratio of the concentration of monomers relative to the concentration of initiator during the course of the polymerisation. Typical chain transfer agents are thiols, halocarbons and cobalt macrocycles.

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The colorant may also comprise a colorant precursor. Colorant may be attached to the water-dissipatable polymer by means of a reaction between a hydroxy group on the polymer with a colorant precursor thereby forming a covalent bond therebetween and subsequently converting the colorant precursor to a colorant. For example the colorant precursor may be converted to a colorant by a process comprising a diazotisation reaction. A diazotisation reaction suitably comprises the steps:

- (i) diazotising an amino group in the colorant precursor using a diazotising agent; and
- (ii) coupling the product of step (i) with a coupling component forming an azo group therebetween.

Suitable colorants include reactive dyes. Reactive dyes are known in the art as dyes as having substituents reactive towards hydroxy groups. These reactive dyes include, but are not limited to: i) chlorine or fluorine containing reactive dyes, for example dichloro and monochloro triazine reactive dyes, ii) vinyl sulphone or protected vinyl sulphone reactive dyes for example sulphato ethyl sulphone reactive dyes, iii) aziridine functionalised dyes, iv) epoxide functionalised dyes, v) isocyanate functionalised dyes and vi) phosphoric acid functionalised dyes which give ester links on dehydration.

The coloured water-dissipatable polymer is preferably prepared by condensing a water-dissipatable polymer carrying hydroxy groups with a colorant having a functional group reactive towards the hydroxy functional groups. Such functional groups are described above. The condensation is performed by preparing a solution or suspension of a water dissipatable polymer carrying hydroxy groups in an aqueous and/or non-aqueous solvent. Preferably the condensation is performed at a pH of 5 to 14, more preferably of 6 to 13, especially of 7 to 12. The condensation is preferably performed in the presence of an inorganic or organic base. Preferred inorganic bases are NaOH, KOH, Na_2CO_3 , K_2CO_3 . Preferred organic bases are trialkyl amines, 1,5-diazabicyclo[4.3.0]non-5-ene (DBN), 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU), and 1,4-diazabicyclo[2.2.2]octane (DABCO). Subsequently an aqueous solution of a colorant is added to the suspension and the mixture is stirred until the reaction is complete.

The coloured water-dissipatable polymer may be isolated from the aqueous system by either acidification and filtration; dilution with a water miscible solvent and filtration; salting out with organic salts or solutions of organic salts or combinations of these methods. Suitable salts include sodium chloride, ammonium chloride, sodium sulphate and lithium chloride. Alternatively the mixture is acidified until the coloured water-dissipatable acrylic polymer precipitates out, or the solvent is evaporated or the solvent is diluted with water until the coloured water-dissipatable polymer precipitates out.

Alternatively the coloured water-dissipatable polymer of the present invention may be purified by ion-exchange methods on cationic resins. Other options include the removal of low molecular weight materials such as co-solvents used for the polymerisation, low molecular weight salts, impurities and free monomers by ultra-filtration, osmosis, reverse osmosis, dialysis, ultra-filtration or a combination thereof, followed by evaporation of the water.

Alternatively a non-aqueous grafting reaction can be used to react the hydroxy-functionalised polymer with a colorant. The colorant and hydroxy-functionalised polymer are stirred in a non-aqueous solvent with a base (either homogeneous or heterogeneous) and heated as required to complete the reaction.

In the case of ester formation, for example reacting an acid functionalised colorant (e.g. phosphoric acid) functionalised and the hydroxy-functionalised polymer, conditions are required to remove the generated water either physically (e.g. by azeotroping, molecular sieves) or chemically using a dehydrating agent such as dicyclohexylcarbodiimide (DCC) or dicyandiamide. Acid catalysts such as p-toluenesulphonic acid or sulphuric acid may also be employed.

In a third aspect of the present invention there is provided an ink comprising the components:

a water-dissipatable polymer according to the first aspect of the present invention; and (b) a liquid medium. Preferably the ink comprises from 0.5 to 50 parts of component (a) and from 50 to 99.5 parts of component (b), wherein all parts are by weight and the number of parts of (a) + (b) = 100. The number of parts of component (a) is preferably from 0.5 to 28, more preferably from 2 to 25, and especially from 2.5 to 20 parts. Preferably component (b) comprises water and an organic solvent. More preferably component (b) comprises from 40 to 95 parts, more preferably from 50 to 90 parts of water; and from 2 to 60 parts, more preferably from 3 to 50 parts, especially from 5 to 35 parts of water-miscible organic solvent. Preferably component (a) is completely dissipated in component (b).

The number of parts of the coloured water-dissipatable polymer is calculated on a 100% solids basis. For example 50g of a 20% solids water-dissipatable polymer is taken as 10g of water-dissipatable polymer.

The inks according to the second aspect of the invention may be prepared by mixing the coloured water-dissipatable polymer with a liquid medium. Suitable techniques are well known in the art, for example agitation, ultrasonication or stirring of the mixture. The mixture of coloured water-dissipatable polymer and liquid medium may be in the form of a dispersion, emulsification, suspension, solution or mixture thereof.

Preferably the coloured water-dissipatable polymer is mixed with a first liquid medium, followed by mixing the resultant mixture with a second liquid medium.

The liquid medium is preferably water, a mixture of water and an organic solvent and an inorganic solvent free from water. For example the coloured water-dissipatable polymer may be added to water followed by the addition of one or more organic solvents. Preferably the first liquid medium is an organic solvent and the second liquid medium is water and a mixture of water and one or more organic solvents.

When the liquid medium comprises a mixture of water and an organic solvent, the weight ratio of water to organic solvent is preferably from 99:1 to 1:99, more preferably from 99:1 to 50:50 and especially from 95:5 to 80:20.

It is preferred that the organic solvent present in the mixture of water and organic solvent is a water-miscible organic solvent or a mixture of such solvents.

Suitable water-miscible organic solvents include C₁₋₅-alkanols, e.g. methanol, ethanol, n-propanol, isopropanol, n-butanol, sec-butanol, tert-butanol and isobutanol; amides, e.g. dimethylformamide and dimethylacetamide; ketones and ketone alcohols, e.g. acetone and diacetone alcohol; C₂₋₄-ether, e.g. tetrahydrofuran and dioxane; alkylene glycols or thioglycols containing a C₂-C₆ alkylene group, e.g. ethylene glycol, propylene glycol, butylene glycol, pentylene glycol and hexylene glycol; poly(alkylene-glycol)s and thioglycol)s, e.g. diethylene glycol, thiodiglycol, polyethylene glycol and polypropylene glycol; polyols, e.g. glycerol and 1,2,6-hexanetriol; and lower alkyl glycol and polyglycol ethers, e.g. 2-methoxyethanol, 2-(2-methoxyethoxy)ethanol, 2-(2-ethoxyethoxy) ethanol, 2-(2-butoxyethoxy)ethanol, 3-butoxypropan-1-ol, 2-[2-(2-methoxyethoxy)-ethoxy]ethanol, 2-[2-(2-ethoxyethoxy)ethoxy]-ethanol; cyclic esters and cyclic amides, e.g. optionally substituted pyrrolidones; sulpholane; and mixtures containing two or more of the aforementioned water-miscible organic solvents. Preferred water-miscible organic solvents are C₁₋₆-alkyl mono ethers of C₂₋₆-alkylene glycols and C₁₋₆-alkyl mono ethers of poly(C₂₋₆-alkylene glycols).

Suitable water-immiscible organic solvents include aromatic hydrocarbons, e.g. toluene, xylene, naphthalene, tetrahydronaphthalene and methyl naphthalene; chlorinated aromatic hydrocarbons, e.g. chlorobenzene, fluorobenzene, chloronaphthalene and bromonaphthalene; esters, e.g. butyl acetate, ethyl acetate, methyl benzoate, ethyl benzoate, benzyl benzoate, butyl benzoate, phenylethyl acetate, butyl lactate, benzyl lactate, diethyleneglycol dipropionate, dimethyl phthalate, diethyl phthalate, dibutyl phthalate, di (2-ethylhexyl) phthalate; alcohols having six or more carbon atoms, e.g. hexanol, octanol, benzyl alcohol, phenyl ethanol, phenoxy ethanol, phenoxy propanol and phenoxy butanol; ethers having at least 5 carbon atoms, preferably C₅₋₁₄ ethers, e.g. anisole and phenetole; nitrocellulose, cellulose ether, cellulose acetate; low odour petroleum distillates; turpentine; white spirits; naphtha; isopropylbiphenyl; terpene; vegetable oil; mineral oil; essential oil; and natural oil; and mixtures of any two or more thereof. Benzyl alcohol is especially preferred.

The ink optionally contains a biocide, for example Proxel GXL. (Proxel is a trade mark of Zeneca Limited) or Kathon (Kathon is a trade mark of Rohm and Haas), a

fungicide, a rheological agent, e.g. a wax (e.g. beeswax), a clay (e.g. bentonite), an IR absorber, for example Projet 900NP (Projet is a trade mark of Zeneca Limited), or a fluorescent brightener, for example C.I. Fluorescent Brightener 179 and/or UV absorber, for example hydroxy phenylbenzotriazole. Furthermore the ink compositions optionally contain a surface active agent, wetting agent and/or an emulsifier, for example those described in McCutcheon's Emulsifiers and Detergents 1996 International Edition or in Surfactants Europa 3rd Edition 1996 each of which is incorporated herein by reference.

The ink preferably has a pH from 3 to 11, more preferably from 4 to 10. The pH selected will depend to some extent on the desired cation for colorant and the materials used to construct the ink jet printer head. The desired pH may be obtained by the addition of an acid, base or a pH buffer. Where a base is used this is preferably the same base as was used to neutralise the anionic dispersing group during the preparation of the water-dissipatable polymer.

The viscosity of the ink is preferably less than 20cp, more preferably less than 15cp, especially less than 10cp, at 20°C.

The inks of the present invention have the advantage that they are suitable not only for the use in piezoelectric ink jet printers but also in thermal and continuous ink jet printers. Many other inks based on polymers work poorly or even not at all in thermal ink jet printers.

Inks of the invention form discrete droplets on the substrate with little tendency for diffusing. Consequently sharp images with excellent print quality and little if any bleed between colours printed side by side can be obtained. Furthermore the inks show good storage stability, wet and light fastness and fastness to both acidic and alkaline highlighter pens.

A further aspect of the invention provides a process for printing an image on a substrate comprising applying thereto an ink according to the first aspect of the present invention by means of an ink jet printer.

The ink jet printer preferably applies the ink to the substrate in the form of droplets which are ejected through a small nozzle onto the substrate. Preferred ink jet printers are piezoelectric ink jet printers and thermal ink jet printers. In thermal ink jet printers, programmed pulses of heat are applied to the ink in a reservoir by means of a resistor adjacent to the nozzle, thereby causing the ink to be ejected in the form of small droplets directed towards the substrate during relative movement between the substrate and the nozzle. In piezoelectric ink jet printers the oscillation of a small crystal causes ejection of the ink from the nozzle.

The substrate is preferably a paper, plastic, or textile material, more preferably a paper, an overhead projector slide or a textile material, especially paper.

Preferred papers are plain, coated or treated papers which may have an acid, alkaline or neutral character. Most preferably the substrate is a coated paper.

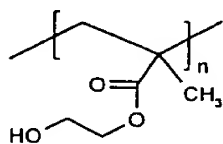
According to a further feature of the invention there is provided an ink jet printer cartridge, optionally refillable, containing an ink as hereinbefore defined.

The invention will now be described by example only. All parts and percentages are by weight unless specified otherwise. In the examples, compounds referred to by reference to CI numbers are the dyestuffs identified by these numbers in the Colour Index International, 3rd Edition, 3rd Revision.

Example 1

Preparation of Water-dissipatable acrylic polymer carrying pendant hydroxyl groups
 MAA/MMA/HEMA/EHA/EA = 15/34/10/10/31 (%W/W)

where Formula 1 represents a repeat unit of 2-hydroxyethylmethacrylate (HEMA) where $n = 10\%$ w/w of the water-dissipatable acrylic polymer.



Formula 1

Stage 1

Table 1

Component Number	Component	Weight (g)
1	Azo-Iso-Butylnitrile (AIBN)	0.6
2	Butyl-3-Mercaptopropionate	7.0
3	Methyl Ethyl ketone	333.5
4	Methyl Acrylic Acid	35.0
5	Methyl Methacrylate	79.6
6	Hydroxy Ethyl Methacrylate	23.3
7	Ethyl Hexyl Acrylate	23.3
8	Ethyl Acrylate	71.9
9	Azo-Iso-Butylnitrile (AIBN)	1.7
10	Methyl Ethyl ketone	30.0

The initial charge (components 1,2,3,4,5,6,7 and 8) were added to a stirred reaction vessel under a nitrogen atmosphere and heated to 80°C. The initiator feed (components 9 and 10) was charged to a 50 ml syringe. At 80°C, the initiator feed was added slowly to the initial charge mixture via the use of a syringe pump such that the total addition time was three hours. When the addition of the initiator feed was completed, the reaction mixture was left to stir for one hour at 90°C. A monomer 'burn-

up' was then carried out by adding V65 initiator 0.2% on solids (V65 = 2,2-azobis(2,4-dimethylvaleronitrile CAS 4419-11-8, V65 is a trade name of Wako) and leaving for a further 40 minutes at 90°C. The product was then cooled to room temperature before being bottled.

5 The solution of the acrylic polymer was found to have a solids content of 40.0%. Molecular weight distribution was done on the acrylic polymer by gel permeation chromatography giving $M_w = 10550$ and $M_n = 4760$.

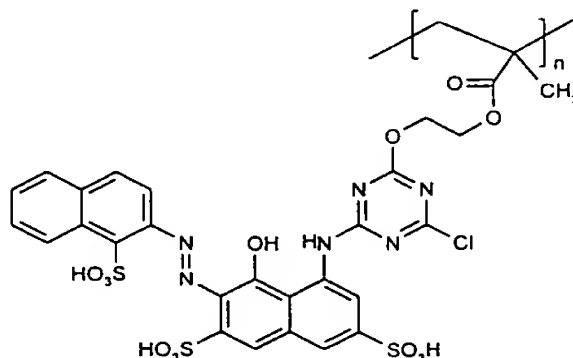
10 Purification of the water dissipatable acrylic polymer was carried out before stage 2 to remove any un-reacted monomer. 10 parts of the acrylic polymer was dissolved in 100 parts of aqueous ammonia (pH 9), cascade filtered and reverse osmosis was carried out until a low conductivity ($<100\mu\text{S}$) was achieved.

Stage 2: Preparation of the coloured water-dissipatable acrylic polymer

15 A suspension of the water-dissipatable acrylic polymer prepared in stage 1 (9 parts) in water (50 parts) at room temperature was adjusted to pH 9.5 by addition of 2N sodium hydroxide solution. The mixture was stirred for 2 hours and then a solution of CI Reactive Red 11 (30 parts) in water (200 parts) was added dropwise over 10 minutes whilst maintaining pH 9.5 by dropwise addition of 2N sodium hydroxide. The solution was left to stir at pH 9.5 and room temperature for 60 hours and then adjusted to pH 1 by addition of excess 2N hydrochloric acid. The resulting solid was isolated by filtration and washed with copious distilled water. The solid was dissolved in aqueous ammonia, precipitated by acidification to pH 1 with 2N hydrochloric acid, re-isolated by filtration and washed with distilled water. The dissolving and precipitation process was then repeated twice.

25 The coloured water-dissipatable acrylic polymer was dissolved in aqueous ammonia and the solution was filtered through a cascade of filters (Glass microfibre GF/A, GF/F and membrane) to 0.45 micron and then purified by reverse osmosis using a 3000 MW cut off membrane and evaporated to dryness at approximately 80°C to give a coloured water-dissipatable acrylic polymer containing a repeat unit of Formula 2:

30 Formula 2



Stage 3: Preparation of Ink

Inks were then prepared by dissolving the specified % of the coloured water-dissipatable acrylic polymer prepared in stage 2 in a stock solution of 9 parts water and 1 part 1-methyl-2-pyrrolidinone with the addition of concentrated ammonia to give a pH of 9-10. The inks were then filtered through a 0.45 micron membrane filter and printed using HP 560 thermal IJ printer. The following results (Table 2) were obtained on Gilbert Bond paper:

Table 2

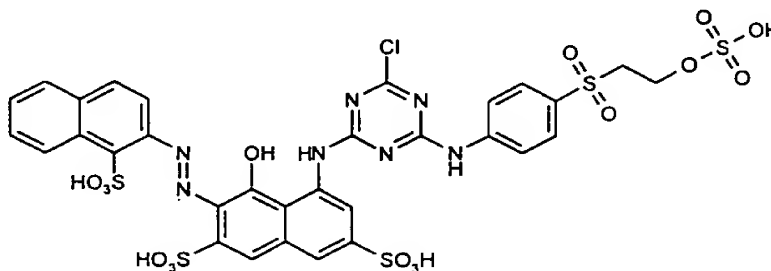
% solid in ink	OD Optical Density	a	b	Run down (5 min)
10	0.362	+33.01	-16.92	10
10	0.720	+54.63	-19.47	9

The polymeric examples have excellent fastness to acidic and alkaline highlighter pens.

Example 2

Stage 1: Preparation of a colorant of formula 3 comprising a dye and a bridging compound

Formula 3



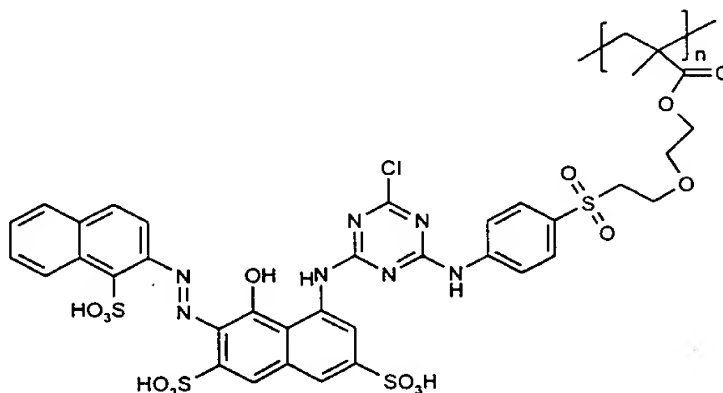
para-Aminobenzenesulfatoethylsulfone (8 parts) was added to a stirred solution of CI Reactive Red II (20 parts) in water (80 parts) at room temperature and the resulting solution was adjusted to pH 7.0 by addition of 2N sodium hydroxide. The solution was allowed to stir at pH 7.0 and room temperature overnight and then salt was added until a solid precipitated. The resulting suspension was filtered and the collected paste was washed with brine.

The coloured paste was dissolved in distilled water and the solution was filtered through a cascade of filters (Glass microfibre GF/A, GF/F and membrane) to 0.45

Stage 2: Preparation of a coloured acrylic polymer

The coloured water-dissipatable acrylic polymer was dissolved in aqueous ammonia and the solution was filtered through a cascade of filters (Glass microfibre GF/A, GF/F and membrane) to 0.45 micron and then purified by reverse osmosis using a 3000 MW cut off membrane and evaporated to dryness at approximately 80°C to give the coloured water-dissipatable acrylic polymer containing a repeat unit of Formula 4:

Formula 4



Inks were then prepared by dissolving the specified % of the coloured water-dissipatable acrylic polymer in a stock solution of 9 parts water and 1 part 1-methyl-2-pyrrolidinone with the addition of concentrated ammonia to give a pH of 9-10. The inks were then filtered through a 0.45 micron membrane filter and printed using HP 560

thermal Ink-Jet printer. The following results (Table 3) were obtained on Gilbert Bond paper

Table 3

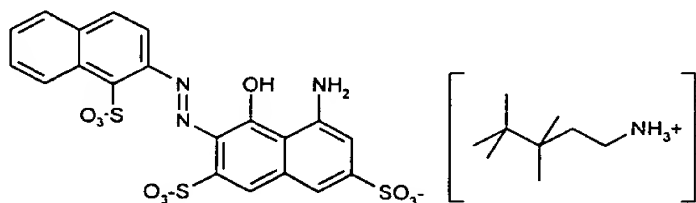
% Solid in Ink	OD	a	b	Run Down (5 min)
10	0.738	+31.07	-17.66	10

The polymeric examples have excellent fastness to acidic and alkaline highlighter pens. The dye lost 5% OD on fading for 100 hours in an accelerated light fastness test.

Example 3

Stage 1: Preparation of iso-nonylamine salt of a colourant of formula 5

Formula 5



iso-Nonylamine (6 parts) was dissolved in distilled water (150 parts) and set stirring. A solution of dye base P-8B sodium salt (5 parts) in water (50 parts) was screened through GF/F filter and added dropwise over 30 minutes to the solution of iso-nonylamine, maintaining pH 7.0 by addition of 2N hydrochloric acid. The mixture was stirred for 30 minutes and then the resulting suspension was filtered. The collected paste was washed with water (20 parts) and then dried at room temperature and under reduced pressure over phosphorus pentaoxide for 24 hours to leave a red solid colorant.

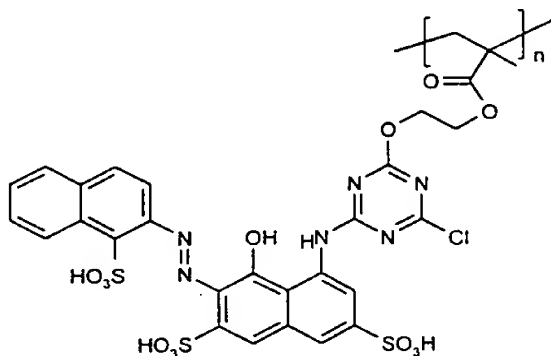
Stage 2: Preparation of coloured acrylic polymer

Cyanuric chloride (1.3 parts) and potassium carbonate (1 part) were added to a stirred solution of the water-dissipatable acrylic polymer prepared in Example 1, stage 1 (10 parts) in tetrahydrofuran (110 parts) at room temperature. The resulting mixture was stirred for 2 hours and then a solution of the colorant prepared in stage 1, (130 parts), triethylamine (1.1 parts) and N,N-dimethylaminopyridine (0.2 parts) in dimethylformamide (260 parts) was added in one portion. The solution was left to stir

for 60 hours and then poured onto vigorously stirred water (3000 parts). The resulting precipitate was collected by filtration and washed with copious distilled water. The solid was dissolved in aqueous ammonia, precipitated by acidification with 2N hydrochloric acid, reisolated by filtration and washed with distilled water. The dissolving and precipitation process was then repeated again.

The coloured water-dissipatable acrylic polymer was dissolved in aqueous ammonia and the solution was filtered through a cascade of filters (Glass microfibre GF/A, GF/F and membrane) to 0.45 micron and then purified by reverse osmosis using a 3000 MW cut off membrane and evaporated to dryness at approximately 80°C to give a coloured water-dissipatable acrylic polymer containing a repeat unit of formula 6:

Formula 6



Stage 3: Preparation of Ink

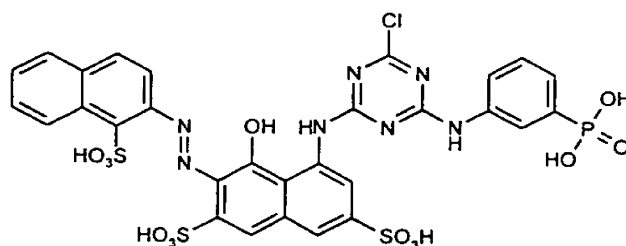
Inks were then prepared by dissolving the specified % of the coloured water-dissipatable acrylic polymer in a stock solution of 9 parts water and 1 part 1-methyl-2-pyrrolidinone with the addition of concentrated ammonia to give a pH of 9-10. The inks were then filtered through a 0.45 micron membrane filter and printed using HP 560 thermal Ink-Jet printer. The following results (Table 4) were obtained on Gilbert Bond paper:

Table 4

% Solid in Ink	OD	a	b	Run Down (5 min)
10	0.393	+30.37	-13.69	10

Example 4 Stage 1: Preparation of a phosphonic acid colorant of formula 7

Formula 7

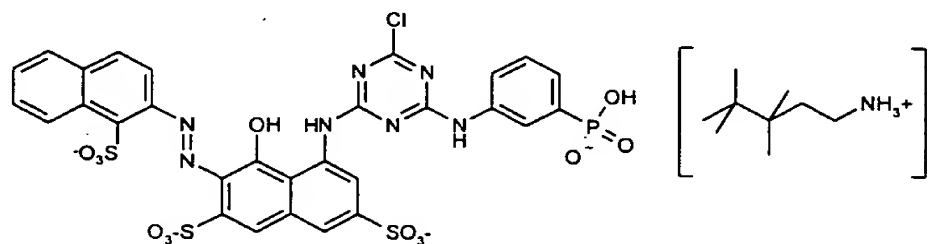


Meta-Aminophenylphosphonic acid (5 parts) was added to a stirred solution of Procion red MX-8B (20 parts) in water (80 parts) at room temperature and the resulting solution was adjusted to pH 7.0 by addition of 2N sodium hydroxide. The solution was allowed to stir at pH 7.0 and room temperature overnight and then salt was added until a solid precipitated. The resulting suspension was filtered and the collected paste was washed with brine.

The coloured paste was dissolved in distilled water and the solution was filtered through a cascade of filters (Glass microfibre GF/A, GF/F and membrane) to 0.45 micron and then purified by reverse osmosis using a 500 MW cut off membrane and evaporated to dryness on a rotary evaporator at 40°C to leave the colorant (9 parts)

Stage 2: Preparation of a iso-nonylamine salt (formula 8) of the phosphonic acid colorant prepared in stage 1

Formula 8



iso-Nonylamine (8 parts) was dissolved in distilled water (150 parts) and set stirring. A solution of the phosphonic acid colorant as prepared in stage 1 (9 parts) in water (70 parts) was screened through GF/F filter and added dropwise over 30 minutes to the solution of iso-nonylamine, maintaining pH 7.0 by addition of 2N hydrochloric acid.

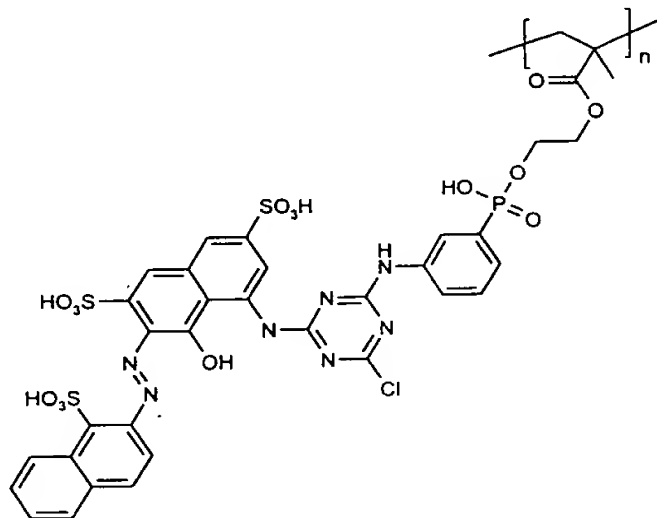
The mixture was stirred for 30 minutes and then the resulting suspension was filtered. The collected paste was washed with water (20 parts) and then dried at room temperature and under reduced pressure over phosphorus pentoxide to leave a red solid (10 parts) colorant.

Stage 3: Preparation of a coloured water-dissipatable acrylic polymer

The water-dissipatable acrylic polymer prepared in Example 1, stage 1 (2 parts), dicyandiamide (1.3 parts) and the colorant prepared in stage 2 (3 parts) were added to dimethylformamide (30 parts) and set stirring. The solution was heated to 130°C for 72 hours and then cooled to room temperature and poured onto water (300 parts). The suspension was adjusted to pH 10 with aqueous ammonia and then the resulting solution was adjusted to pH 1 by addition of 2N hydrochloric acid. The supernatant liquid was decanted and the resulting tar-like material was dissolved in acetone (30 parts) and diluted with water (300 parts).

The suspension was adjusted to pH 10 with aqueous ammonia and the solution was filtered through a cascade of filters (Glass microfibre GF/A, GF/F and membrane) to 0.45 micron and then purified by reverse osmosis using a 3000 MW cut off membrane and evaporated to dryness at approximately 80°C to give the coloured water-dissipatable acrylic polymer containing a repeat unit of formula 9:

Formula 9



Stage 4: Preparation of ink

Inks were then prepared by dissolving the specified % of the coloured acrylic polymer in a stock solution of 9 parts water and 1 part 1-methyl-2-pyrrolidinone with the

addition of concentrated ammonia to give a pH of 9-10. The inks were then filtered through a 0.45 micron membrane filter and printed using HP 560 thermal Ink-Jet printer. The following results (Table 5) were obtained on Gilbert Bond paper:

5

Table 5

% Solid in Ink	OD	a	b	Run Down (5 min)
10	0.865	+53.95	-15.91	9

Example 5

Further inks may be prepared having the formulations described in Tables 2 and 3 below wherein the following abbreviations are used. The number of parts by weight of each component is given. Water is included in each formulation to make the total number of parts up to 100. These inks may be applied to plain paper using an Ink-Jet printer.

FRU : fructose

PU* : Identifies which of the acrylic polymers as prepared in Examples 1 and 2 may be used. The number of parts by weight of PU is shown in brackets.

BZ : Benzyl alcohol

DEG : Diethylene glycol

DMB : Diethyleneglycol monobutyl ether

ACE : Acetone

IPA : Isopropyl alcohol

MEOH : Methanol

2P : 2-Pyrrolidone

MIBK : Methylisobutyl ketone

SUR : Surfinol 465 (a surfactant)

PHO : K_2PO_4

TEN : triethanolamine

NMP : N-methylpyrrolidone

TDG : Thiodiglycol

CAP : Caprolactam

BUT : Butylcellosolve

GLY : Glycerol

Colorant 1 : Coloured acrylic polymer as prepared in Examples 1.

Colorant 2 : Coloured acrylic polymer as prepared in Examples 2.

Colorant 3 : Coloured acrylic polymer as prepared in Examples 3.

Colorant 4 : Coloured acrylic polymer as prepared in Examples 4.

TABLE 2

Colorant	Colorant Content	BZ	DEG	ACE	NaOH	(NH ₄) ₂ SO ₄	IPA	MEOH	2P	MIBK	BUT
1	2	4	6						10	10	
2	3	5	5		0.2				15		
3	4	8							20	1	3
4	4	8			0.5		6		8	4	1
1+2	(4+4)	15	3	3					5	4	
1	5	20					8				
2	3	4		5				6	20	5	1
3	4	5	2	10		0.3					
4	3	6	6	6			5	4	6	5	
4	5	6	2	2			1		4		
2	2	5							10		4
1	4	5		5		0.1		2		6	3
3	2.0	15		10	0.3		3		10		
2	6	20	2	1					15	3	
1	1.0	5	4						20	5	

TABLE 3

Colorant	Colorant Content	BZ	NMP	SUR	TEN	TDG	FRU	PHO	DMB	CH ₃ NH ₂	CAP
4	2	5		0.15	0.4	20					
1	3	6	15					0.05			4
1	4	10		0.3		15				0.2	
3	1	10	20				0.5	0.2			2
2	8	15	15		1	5					
1	5	10	10	0.2			1		4	0.2	
1	3	5	3				2		6		
4	10	9	7		0.5			1	5		
2	5	11				12			8		1
3	6	5	17						7		
1	2	8	5	0.1	0.2	2	0.5	0.1	5		
2	2	10	5			10			5		2
4	8	5	8						9		
3	10	4							12		
1	10	10	1		1	1				1	1

CLAIMS

1. A water-dissipatable polymer having colorant attached thereto through a covalent -O- link.

2. A water-dissipatable acrylic polymer according to claim 1.

3. A water-dissipatable polymer according to claim 1 or 2 wherein the polymer has been obtained from the polymerisation of one or more olefinically unsaturated monomers having water dispersing groups, and one or more olefinically unsaturated monomers having hydroxyl functional groups optionally in the presence of one or more olefinically unsaturated monomers which are free from water dispersing groups.

4. A water-dissipatable polymer according to claim 1 or 2 wherein the colorant is attached thereto by means of a reaction between a hydroxy group on the polymer with a colorant having a functional group capable of reacting with the hydroxy group.

5. A water-dissipatable polymer according to claim 1 or 2 wherein the colorant is attached thereto by means of a reaction between a hydroxy group on the polymer with a colorant precursor thereby forming a covalent bond therebetween and subsequently converting the colorant precursor to a colorant

6. A water-dissipatable polymer according to claim 1 or 2 wherein the colorant is attached thereto by means of a reaction between a hydroxy group on the polymer with a bridging compound thereby forming a covalent bond therebetween and subsequently reacting the bridging compound with a colorant or colorant precursor.

7. A water-dissipatable polymer according to claim 5 wherein the colorant precursor is converted to a colorant by a process comprising a diazotisation reaction.

8. A water-dissipatable polymer according to claim 7 wherein the diazotisation reaction comprises the steps:

- (i) diazotising an amino group in the colorant precursor using a diazotising agent; and
- (ii) coupling the product of step (i) with a coupling component forming an azo group therebetween.

9. A water-dissipatable polymer according to any one of the preceding claims having a Mn less than 25,000.

10. An ink comprising the components:

- (a) a water-dissipatable polymer according to any one of claims 1 to 7; and
(b) a liquid medium.

5 11. An ink according to claim 10 wherein component (a) is completely dissipated in component (b).

10 12. An ink according to any one of claims 8 to 11 which comprises from 0.5 to 50 parts of component (a) and from 50 to 99.5 parts of component (b), wherein all parts are by weight the number of parts of (a) + (b) = 100.

13. An ink according to claim 10, 11 or 12 wherein component (b) comprises water and an organic solvent.

15 14. An ink according to claim 13 where component (b) comprises from 40 to 95 parts of water and from 2 to 60 parts of water-miscible organic solvent.

15. An ink according to any one of claims 10 to 14 having a viscosity less than 20 cp at 20°C.

20 16. An ink according to any one of claims 10 to 15 for use in an ink jet printer.

17. A process for forming an image on a substrate comprising applying thereto an ink using an ink jet printer, characterised in that the ink is as defined in any one of claims 10 to 15.

25 18. A paper or an overhead projector slide printed with an ink as defined in any one of claims 10 to 15.

30 19. An ink jet printer cartridge, optionally refillable, containing an ink as defined in any one of claims 10 to 15.

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